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HARDNESS IMPROVEMENT AND LUBRICATION OF ANODIZED AA7075 WITH POLYTETRAFLUOROETHYLENE ADDITIVE

Anodizing is widely employed to improve the corrosion resistance of aluminum. However, to maximize its industrial applicability, further enhancement of the hardness of the anodic oxide film is required. In this context, polymeric materials have been commonly incorporated to improve the hardness of the anodic film while also imparting lubricating properties. In this study, anodizing conditions were established based on temperature (5°C and 15°C) and time (15 min and 30 min), then polytetrafluoroethylene (PTFE) was added during the anodizing process to enhance hardness and provide lubrication. The morphology of the anodic oxide film and the co-deposition of PTFE were confirmed using FE-SEM/EDS and FT-IR analyses. The co-deposition of PTFE appears to contribute not only to improved lubricity but also to increased densification of the anodic oxide film. Based on this inference, the study can be extended to explore various anodic oxide film configurations with enhanced hardness and lubricating properties by optimizing parameters such as temperature, anodizing time, and the combination of additional additives.

Keywords: AA7075 aluminum; anodizing; polytetrafluoroethylene (PTFE); Lubricant; Friction coefficient

1. Introduction

Anodizing is one of the most widely used methods to improve the corrosion resistance of aluminum (Al). To further enhance hardness or impart lubricity, several studies have employed post-treatments in which anodized films are immersed in or coated with polymer solutions [1]. In particular, polymers such as MoS₂ and PTFE have been utilized to improve lubricity [2]. Previous studies have investigated the improvement of wear resistance through friction coefficient control using PTFE coatings; however, such approaches are limited by the confinement of PTFE distribution only to the surface layer. Therefore, in this work, PTFE particles were dispersed in the anodizing solution, enabling their co-deposition throughout the entire oxide layer, including the pores [3]. In this study, optimal anodizing conditions were established to maximize hardness with respect to temperature and time. Subsequently, it was found to the addition of PTFE to the sulfuric acid solution used for anodizing improved the surface lubricity and increased the hardness of Al 7075.

2. Experimental

2.1. Sample fabrication

AA7075 substrates with dimensions of 25 mm × 25 mm were initially subjected to alkaline cleaning for 80 seconds. Subsequently, degreasing was performed in a mixed solution of nitric acid and Alclean for 180 seconds. A desmut process was then carried out in a nitric acid (HNO₃) solution for 120 seconds. Following this, anodizing was conducted in a sulfuric acid solution using a platinum cathode. For PTFE additive incorporation, PTFE was added to the sulfuric acid solution (240 g/L) at concentrations of 1, 3, and 5 g/L. Anodizing was performed by gradually increasing the voltage from 5 V to 14 V in 30-second intervals, after which the voltage was maintained at 14 V. During the anodizing process, the electrolyte was stirred at 300 rpm.

2.2. Sample analysis

After anodizing, the specimens were coated with platinum and the surface morphology was observed using a field emission

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scanning electron microscope (FE-SEM, JSM-IT700HR, JEOL, Japan). The surface morphology of the bare and anodized AA7075 specimen was obtained using a secondary electron detector at 15 kV. The presence of PTFE coating on the anodized surface was confirmed using energy-dispersive X-ray spectroscopy (EDS) and Fourier-transform infrared spectroscopy (FT-IR, Nicolet iS20, Thermo-Scientific, USA), and glow discharge optical emission spectrometer (GD-OES, GD-profiler 2, Horiba, Japan). The glossiness of the anodized surface was measured using a gloss meter (BYK micro-TRI-gloss). Microhardness was evaluated using a micro-Vickers hardness tester. Friction coefficient testing was conducted in accordance with the ISO 8295 standard.

3. Results and discussion

Fig. 1 shows the FE-SEM images of the surface of Bare AA 7075 without anodizing and AA 7075 specimens anodized at different temperatures and times. Since the Bare AA 7075 specimen was not anodized, only a negligible presence of pores can be observed on its surface (Fig. 1(a)). In contrast, the anodized AA 7075 specimens exhibit a porous surface structure. When anodizing was conducted at a relatively low temperature of 5°C (Fig. 1(b), 1(c)), the pore size was smaller than that of specimens anodized at 15°C.

This result is consistent with the previous reports which suggest a trend that the pore size tends to increase with increasing anodizing temperature [1].

Fig. 2 illustrates the variations in anodic oxide film thickness and hardness with anodizing temperature, as well as the changes in glossiness depending on anodizing temperature and time. As the anodizing temperature increases, the anodic oxide film became thicker (Fig. 2(a)), which is likely attributable to the temperature-dependent reaction rate [2]. The faster reaction rate

can be linked to the more rapid formation of pores, as observed in Fig. 1. In addition, the hardness of the anodic oxide film also increases with rising anodizing temperature (Fig. 2(b)). This may seem contradictory to the observation in Fig. 1, where the pore size increases at higher temperatures. However, this increase in hardness can be explained by the denser vertical growth of the anodic oxide film at elevated temperatures [4]. Furthermore, the glossiness was also found to increase with anodizing temperature (Fig. 2(c)), which is presumed to result from the reduced surface roughness of the anodic oxide layer [5].

Fig. 3 presents the SEM image of the AA7075 specimen anodized without PTFE addition (Fig. 3(a)), and the SEM image (Fig. 3(b)), EDS spectrum (Fig. 3(c)), and FT-IR analysis (Fig. 3(d)) of the AA7075 specimen anodized with PTFE added to the electrolyte. When anodizing was conducted with PTFE addition, white particles – presumed to be PTFE agglomerates – were observed on the surface (Fig. 3(b)). The EDS spectrum confirmed the presence of carbon (C) and fluorine (F) elements, which are constituents of the PTFE molecule (Fig. 3(c)). Additionally, the FT-IR results also show the characteristic absorption peak corresponding to the $-CF_2$ bonding, providing additional evidence the incorporation of PTFE into the anodic oxide film (Fig. 3(d)). According to the GD-OES results, the bare AA7075 specimen exhibited exposure of the Al substrate following the natural oxide film, whereas the anodized specimen with PTFE addition demonstrated the formation of a well-developed Al_2O_3 layer (Fig. 3(e), 3(f)). In the case of the anodized film formed with PTFE additives, almost no carbon signal was detected within the oxide layer region (Fig. 3(f)). Taken together with the FT-IR and GD-OES results, it can be inferred that PTFE particles are likely distributed in the form of nanoscale co-deposits within the anodic pores and the surface of the anodized film. Fig. 4 illustrates the changes in hardness (Fig. 4(a)) and friction coefficient (Fig. 4(b)) of anodized AA 7075 with varying concentrations

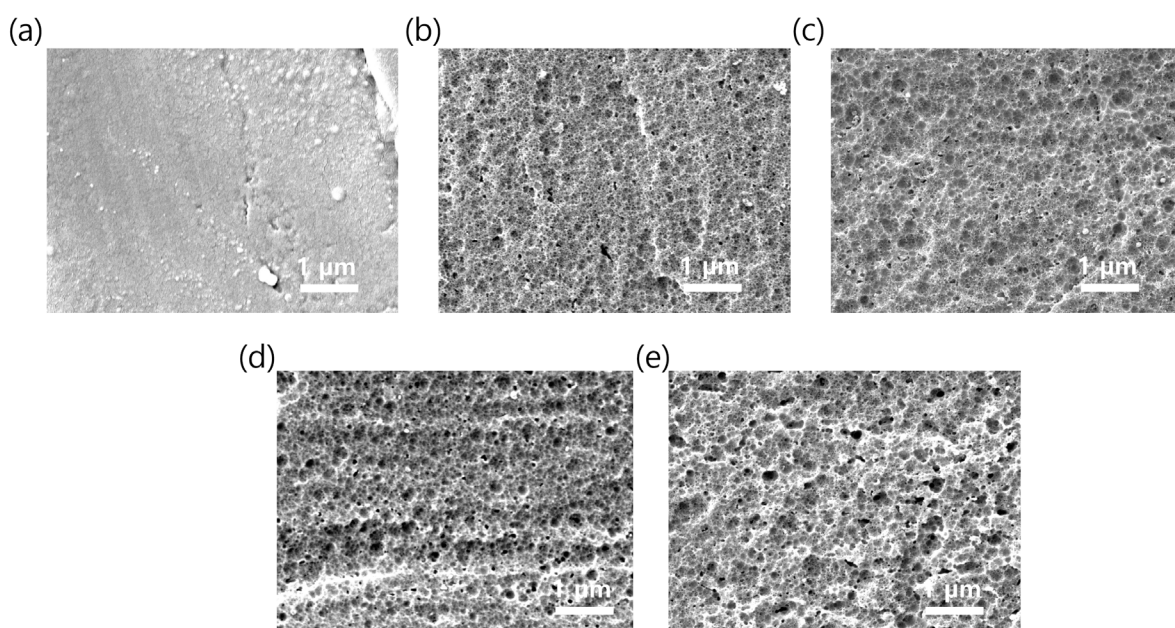


Fig. 1. Anodized AA 7075 with different temperature (5°C / 15°C) and time (15 min / 30 min). (a) Bare AA 7075, (b)–(e) Anodized AA 7075 with 5°C 15 min, 5°C 30 min, 15°C 15 min, 15°C 30 min, respectively

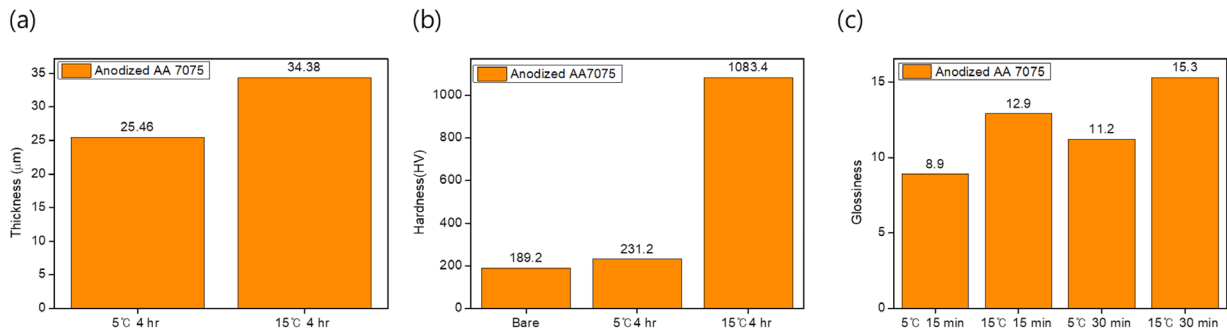


Fig. 2. Thickness, Hardness, and Glossiness of Bare and anodized AA7075 with temperature and time variation. (a) Thickness comparison of anodized AA7075 for 4 hr with different temperature, (b) Hardness comparison of Bare and anodized AA7075 for 4 hr with different temperature, (c) Glossiness comparison of anodized AA7075 with different temperature and time.

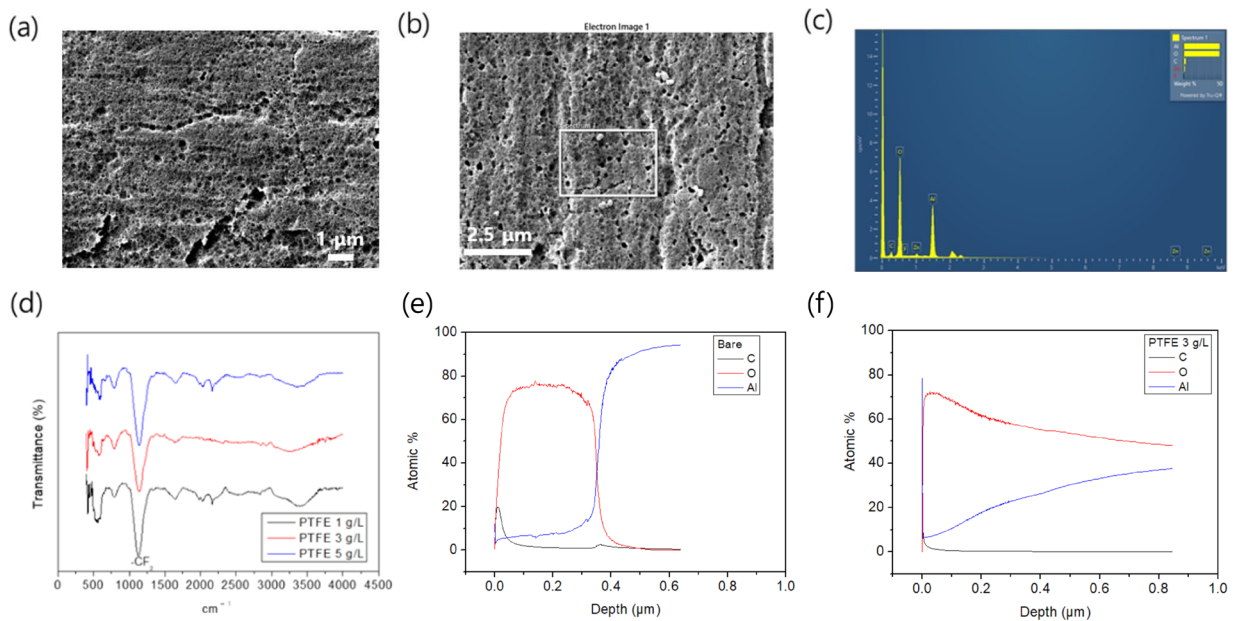


Fig. 3. SEM image of anodized AA 7075 without PTFE (a), with PTFE additive (b), EDS pattern of anodized film with PTFE additive (c), FT-IR data anodized AA 7075 with PTFE(d), GD-OES data of Bare AA7075(e), and of anodized AA7075 with PTFE(f)

of PTFE additives. The enhancement in hardness due to PTFE addition is presumed to result from the dispersion of PTFE particles between anodizing pores, which reduces the inter-particle distance and contributes to increased hardness [6]. In addition, the distribution of PTFE particles around the pores, together with their pinning effect at grain boundaries, is considered to

contribute to hardness enhancement by increasing resistance to grain boundary migration [7]. In addition, the incorporation of PTFE – being relatively softer than Al_2O_3 is considered to induce a stress-distribution effect. This stress distribution likely contributes to the observed reduction in the friction coefficient, as shown in Fig. 4(b). Furthermore, PTFE itself possesses

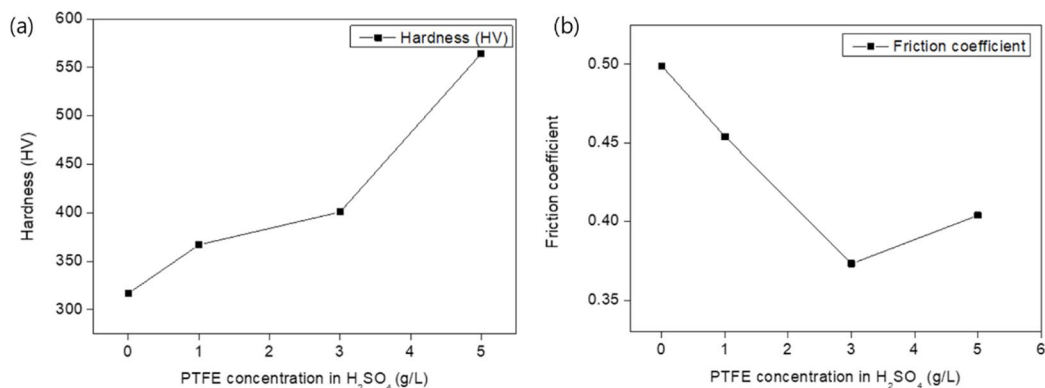


Fig. 4. Tribological property of anodized AA 7075 in sulfuric acid solution with and without PTFE additive (1/3/5 g/L)

inherent lubricating properties, Therefore, during the friction process, PTFE particles distributed within the pores and on the surface can form a thin lubricating film, thereby reducing the coefficient of friction [3,8]; thus, the overall coating of the anodic oxide film with PTFE is believed to improve the wear resistance of the material.

4. Conclusion

In this study, the addition of PTFE to a sulfuric acid-based anodizing solution was found to enhance both the hardness and lubricating properties of the anodized film on AA7075 alloy. The incorporation of PTFE into the anodizing solution is presumed to fill the pores of the anodic oxide layer, leading to a reduction in inter-particle distance, which contributes to increased hardness. However, due to the quantitative detection limit of PTFE, further TEM investigations are required to clarify its role in grain boundary pinning. Additionally, the presence of PTFE is believed to facilitate stress distribution, thereby reducing the coefficient of friction.

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